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# THE DEVELOPMENT OF DISPERSION-STRENGTHENED NICKEL-BASE CORROSION-RESISTANT ALLOYS

by

STAFF MEMBERS  
MELPAR MATERIALS LABORATORY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY REPORT

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NICKEL-BASE CORROSION-RESISTANT ALLOYS

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May 1967

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THE DEVELOPMENT OF DISPERSION-STRENGTHENED  
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ABSTRACT

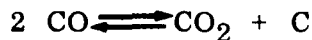
The results of a program to produce a dispersion-strengthened 70 nickel—20 chromium—10 molybdenum alloy by vapor plating in a fluidized bed of oxide particles are described. The nickel vapor was obtained by thermally decomposing nickel carbonyl,  $\text{Ni}(\text{CO})_4$ . The chromium vapor was obtained by decomposing dicumene chromium,  $\text{Cr}(\text{C}_9\text{H}_{12})_2$ , and the molybdenum vapor was obtained by reducing molybdenum pentachloride,  $\text{MoCl}_5$ . Single and multi-element alloys were made. Metallographic examination of single-element deposit agglomerates of chromium or nickel showed a uniform particle distribution. Production of the triple-element dispersion-strengthened alloys by vapor deposition in a fluidized bed proved unsuccessful because of the low plating rates and difficulties in controlling the chemistry of the product. The triple alloys were characterized by unusually high carbon contents (5 wt percent), owing chiefly to the apparent decomposition of the carbonyl radical and carbon monoxide carrier gas used in vapor plating the nickel. The triple-element matrix alloys were also characterized by the presence of agglomerated oxide particles. These studies indicate that it is possible to vapor plate submicron oxide particles in a fluidized bed with either nickel or chromium and to obtain uniform fine oxide dispersions. The results of adding the nickel to the alloy by an alternative method are also discussed.

# THE DEVELOPMENT OF DISPERSION-STRENGTHENED NICKEL-BASE CORROSION-RESISTANT ALLOYS

by Staff Members  
Melpar Materials Laboratory

## SUMMARY

Attempts were made to produce a 70 nickel—20 chromium—10 molybdenum dispersion-strengthened alloy by plating 0.1-micron-diameter zirconia particles successively with molybdenum, chromium and nickel. This plating sequence was in the order of decreasing decomposition temperature of the precursor compounds. The molybdenum was deposited by decomposing  $\text{MoCl}_5$  in the vapor state at  $575^\circ\text{C}$ . During this process, the zirconia was observed to agglomerate and sinter into approximately 25-micron-diameter particles. Another major problem in building up the three-element alloy was in the deposition of the nickel. The nickel was deposited by thermally decomposing nickel-tetracarbonyl,  $\text{Ni}(\text{CO})_4$ . When nickel was deposited on the chromium layer for making the three-element alloy, the following reaction took place:



The excess carbon monoxide,  $\text{CO}$ , came from both the carrier gas and the decomposing nickel carbonyl. Alloys produced in this manner contained more than 5 wt percent carbon.

Attempts were made to add submicron nickel powder to the fluidized bed after plating the molybdenum and the chromium by the vapor deposition process. These procedures did not produce the desired degree of dispersoid distribution and elemental homogeneity, owing partly to the agglomeration of the fine-particle oxide particle during molybdenum deposition.

Vapor plating in a fluidized bed has produced uniform fine oxide dispersions with either nickel or chromium. The fluidized-bed process is capable of producing dispersion-strengthened alloys having good dispersoid distribution, provided the precursor compounds used for plating the elements decompose at low temperatures and that the oxide dispersoids do not agglomerate.

## INTRODUCTION

The development of airbreathing engines of higher thrust is limited chiefly by the properties of the materials used for the turbine stator vanes and the rotating turbine buckets. One of the most promising ways to develop alloys with higher use temperatures and stresses is through dispersion strengthening. At present, the most promising dispersion-strengthened materials appear to be such dispersion-strengthened nickel base alloys as have been produced by DuPont and Sheritt-Gordon.

It was a goal of this program to produce a material with a 3,000 hour rupture strength of 15,000 psi at 2000° F. This was to be accomplished with an alloy that combines both dispersion and solid-solution strengthening. An alloy with 2 to 8 vol percent dispersoid was to be produced with a matrix composition of 70 Ni — 20 Cr — 10 Mo.

The addition of chromium serves two purposes: it greatly increases the oxidation resistance of nickel and, it acts as a solid-solution strengthener of nickel. Molybdenum is one of the most effective solid-solution strengtheners of nickel and was added for this reason. Thus, a dispersion-strengthened alloy with a matrix of nickel containing chromium and molybdenum might meet the stringent mechanical strength and oxidation resistance specifications.

Effective dispersion strengthening requires that the second-phase dispersoid particles be uniformly distributed. The strength of dispersion-strengthened alloys is proportional to  $d^{-1/2}$  for coarse particles and  $d^{-1}$  for fine particles,<sup>(1, 2)</sup> where  $d$  is the mean free path between oxide particles. To optimize strength and ductility, it is necessary to use the smallest particles possible consistent with adequate dispersoid-matrix stability. This permits the smallest mean free path,  $d$ , with a low volume fraction of dispersed phase.

To accomplish these goals it was postulated that if fine oxide particles ( $< 0.1$  micron diameter) could be uniformly plated with matrix elements, an ideal dispersion could be produced. The mean free path,  $d$ , would be minimized and the particle spacing would be uniform. To effect the uniform plating of the particles, a bed of fine oxide particles was fluidized with a gas stream. By decomposing the various volatile metallic precursor compounds in the region of the fluidized particles, the metal(s) could then be plated out on the oxide particles. This vapor plating method had previously been proven feasible for producing dispersion-hardened gold and platinum alloys.<sup>(3)</sup>

Previous investigators of chemical vapor deposition<sup>(4)</sup> have relied almost exclusively upon nickel tetracarbonyl for producing nickel coatings. They showed that molybdenum coatings could be prepared by chemical vapor deposited from both  $\text{MoCl}_5$  and the molybdenum carbonyl. The molybdenum carbonyl may be decomposed at lower temperatures (400° vs 650° C) than the molybdenum pentachloride but carbon contamination has been a problem in coatings produced from the carbonyl. For this reason,

molybdenum pentachloride was selected as the precursor compound to produce molybdenum coatings. Chromium coatings have been produced by decomposing chromium iodide, chromium carbonyl, and dicumene chromium<sup>(4)</sup>. Dicumene chromium decomposes at the lowest temperature of these three materials and was selected for this reason.

It was the purpose of Task I of this program to produce dispersion-strengthened billets of a corrosion-resistant nickel-base matrix containing three levels of each of two oxide dispersoids. The target alloys contained 70 Ni — 20 Cr — 10 Mo with 2, 5 or 7 volume percent ThO<sub>2</sub> or ZrO<sub>2</sub>. The average oxide dispersoid was to be  $\leq 0.02$  to 0.10 microns with an interparticle spacing in the matrix of  $\leq 0.3$  to 1.0 microns. Vapor plating of the oxide dispersoids was to be conducted in a fluidized bed of the dispersoid particles to produce a multilayer coating of Ni, Cr, and Mo. Coated particle agglomeration was to be accomplished by collapsing the fluidized bed and raising the temperature while maintaining a reducing atmosphere. The billets were to be made by isostatically pressing the agglomerated vapor coated dispersoid particles.

Evaluation of the material so produced was to include metallography by both optical and electron microscopy, thermal stability tests and chemical analysis. Linear analysis of the electron photomicrographs was to be used to determine the microstructural parameters. Thermal stability was to be determined by a 100-hour heat treatment in an inert atmosphere at a temperature equivalent to 0.85 of the melting point. Linear analysis was to be carried out after heat treatment to determine any microstructural changes.

It was the purpose of task II of this program to hot extrude the billets produced in task I, and to obtain mechanical property data for the material.

## EXPERIMENTAL PROCEDURE

### Design of the Fluidized-Bed System

The first objective of this program was to design a system in which the fine particle dispersoids could be fluidized and subsequently vapor plated. The design for the fluidized bed system as shown in figures 1 and 2 evolved from a series of experiments to obtain optimum deposition with minimum blowover. A controlling part of the design is the bottom of the fluidized bed.

The first design shown in figure 3-a proved impractical because the glass frit became plugged by the decomposing compounds. For this reason, a tapered column was used. The taper designs shown in figures 3-b and 3-c permitted the oxide powder to fall into the "T" and cause plugging. By extending the taper section and placing an

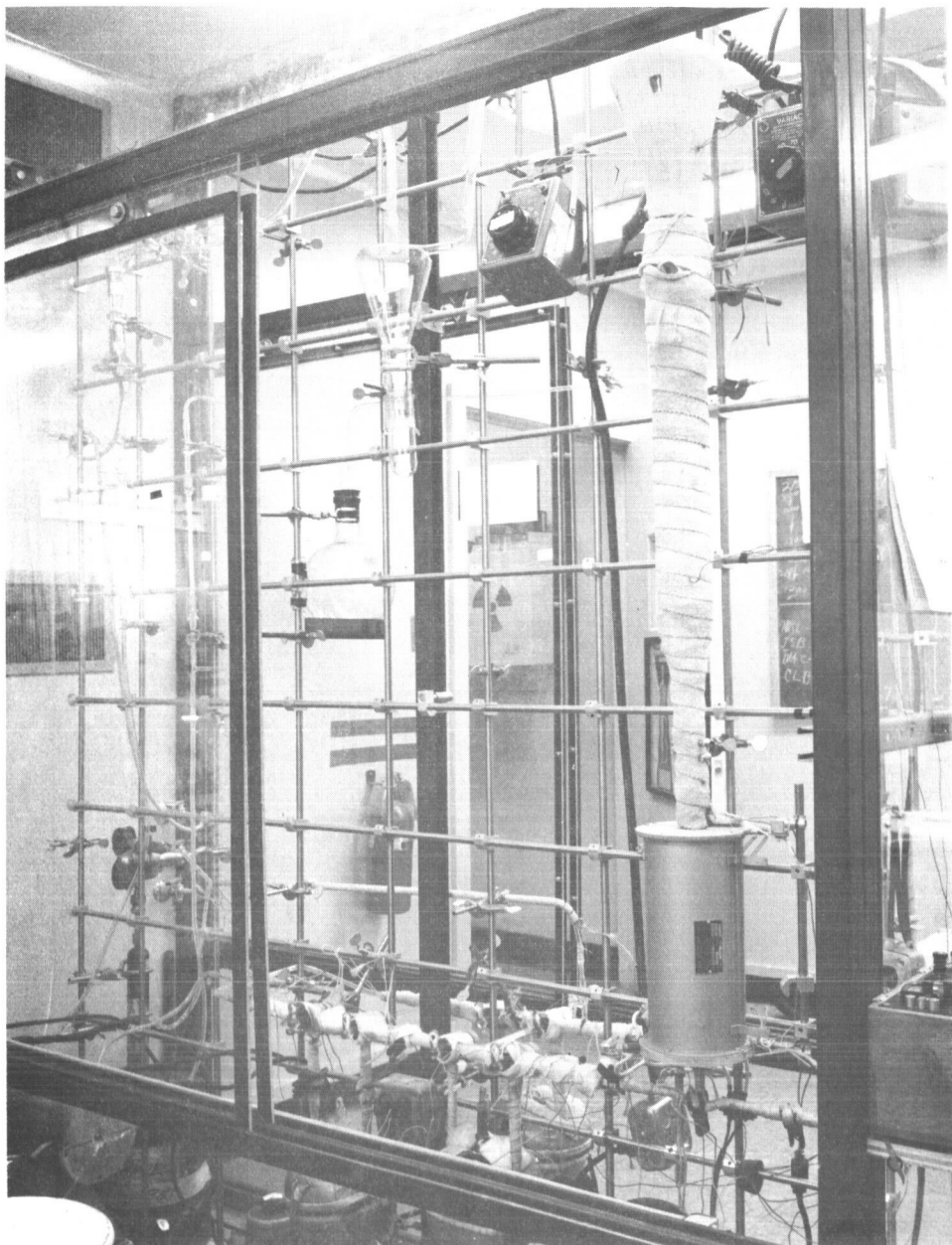


Figure 1. Fluidized-Bed Vapor Plating System for Producing Dispersion-Hardened Alloys



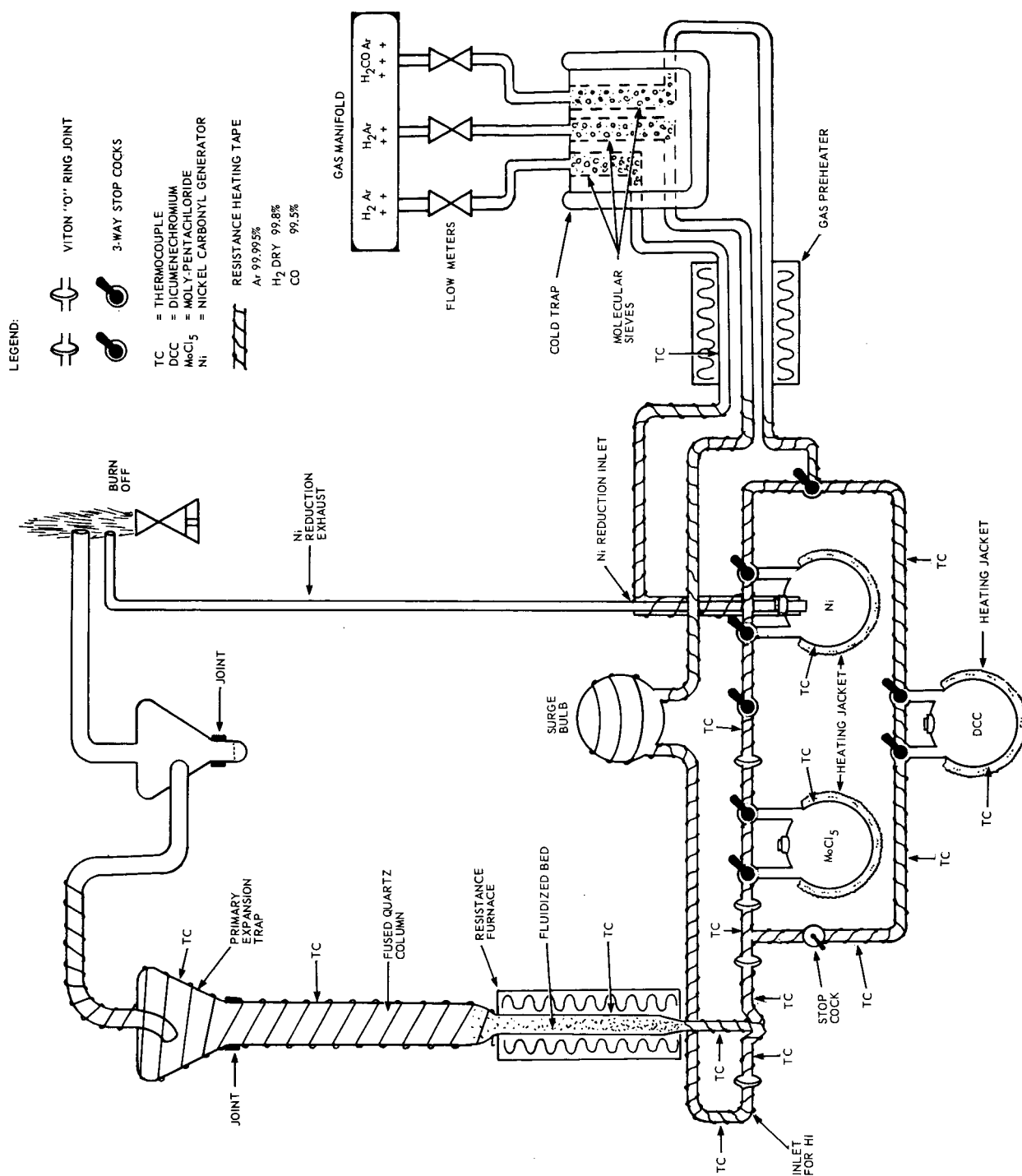


Figure 2. Fluidized-Bed Reactor for Producing Dispersion-Hardened Alloys

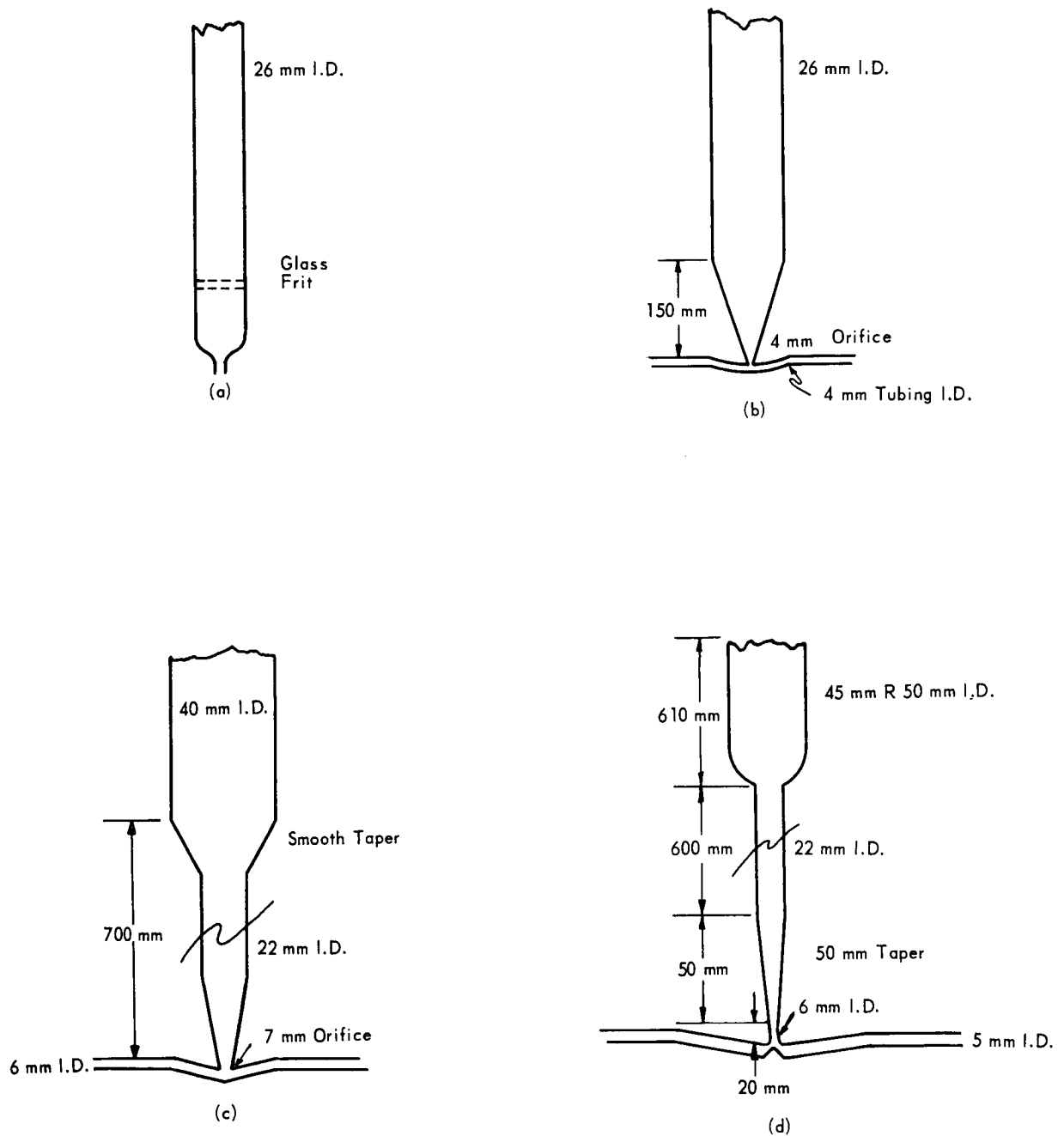


Figure 3. Designs for Bottom Part of Column of Fluidized Bed

undulation at the base of the column, plugging was minimized. This design is shown in figure 3-d. The sizes of the column shown in figure 3-d are optimized and identical with those used in most of the experiments. Orifices larger than 6-mm ID permitted oxide from the bed to run into the "T" and eventually cause plugging.

To minimize blowover, the column was expanded from 45-mm I. D. to 70-mm I. D. above the heated zone. Above the expanded section an inverted Erlenmeyer flask was attached to the column, as shown in figure 1.

It was necessary to heat all of the gas lines with heating tape as shown in figure 1 to prevent condensation of the volatile components.

From the observation of the operation of tapered fluidized beds, the following generalizations were made:

- a. To obtain turbulent fluidization, a critical amount of dispersoid power (for example, 7 g of MgO for a 2-in. -dia. column)<sup>(5)</sup> must be used together with a critical flow rate.
- b. A vibrator was necessary to prevent sticking of the powders to the heated walls.
- c. Too large an orifice at the bottom permitted powder to enter the "T", which resulted in plugging.
- d. Too narrow an orifice at the bottom caused the gas flow to channel through the bed instead of fluidizing the bed. The particle bed would ultimately collapse in this condition.

#### Fluidized Bed Reactor Operation

In actual practice, the operation of the fluidized-bed system shown in figures 1 and 2 was as follows. Gas from cylinders passed through a cold trap and then through a preheat furnace. One line of the gas was fed into one side of the "T" leading to the fluidized bed and served to support the bed of fine particle dispersoids. The other line of gas passed through the precursor compound. After the vapor plating operation was completed, it was routine to raise the temperature of the fluidized bed 50° to 200° C over the decomposition temperature of the last element deposited for 1 to 2 hours. This caused the individual coated dispersoid particles to agglomerate into 25- to 50-micron-diameter composite particles. These large particles were not pyrophoric and could be easily handled in air.

## Materials Used for Vapor Plating

Dispersoid Material.— Two dispersoid materials, thoria and zirconia, were used on this program. Both were supplied by Vitro Laboratories. Examination by electron microscopy showed both materials to be less than 0.1 micron in diameter. The average particle size appeared to be 0.02 to 0.05 microns with less than 5 percent of the particles being 0.3-0.7 microns in diameter.

Nickel Plating Materials.— The nickel used for producing the nickel carbonyl was Fisher low cobalt reagent grade -200 mesh. Chemically pure grade carbon monoxide was used for preparing the nickel tetracarbonyl. High-purity dry hydrogen and 99.995 percent pure argon were used in all of the plating runs.

The nickel added directly to molybdenum and chromium plated zirconia was Sheritt-Gordon NF-IM (0.6 to 1.4 micron) powder.

Chromium Plating Materials.— The dicumene chromium was obtained from Union Carbide Chemical Company (90 percent pure).

Molybdenum Plating Materials. — The molybdenum pentachloride obtained from Climax Molybdenum Co. had a batch analysis as follows: Mo 35.1 percent, Cl 63.9 percent, Fe 0.042 percent, Si 0.006 percent, Cu 0.0007 percent, Ni 0.012 percent.

## Nickel Deposition

Nickel coatings were produced by the decomposition of nickel tetracarbonyl,  $\text{Ni}(\text{CO})_4$ . Nickel tetracarbonyl was prepared continuously in the system. Hydrogen gas was passed through high-purity nickel powder, which was heated to 400° C, to reduce any surface oxide and to activate the surface. After reducing with hydrogen for 4 hours, carbon monoxide was passed into the nickel at 125° C and a flow rate of one liter/min. The carbon monoxide reacts readily at this temperature to form nickel carbonyl,  $\text{Ni}(\text{CO})_4$ .

The nickel carbonyl then passed through feeder lines heated to 100° C and into the fluidized bed column heated to a temperature between 160° C and 210° C. The nickel tetracarbonyl decomposes according to the reaction



The nickel plates out on the fine particles and the CO passes out of the system to a burn-off. Under these conditions, the plating rate was 0.50 g/hr.

## Chromium Deposition

The chromium deposition techniques were developed as part of another investigation. (5)

The chromium was deposited by decomposing dicumene chromium,  $\text{Cr}(\text{C}_9\text{H}_{12})_2$ . Hydrogen and argon were mixed in 1:1 ratio and passed into a container of dicumene chromium heated to  $190^\circ\text{C}$ . The dicumene chromium vapor pressure at this temperature is 10 torrs. The vapor and carrier gases then passed through feeder lines at  $200^\circ\text{C}$  and the vapor was decomposed in the fluidized bed at  $325^\circ\text{C}$ . Temperatures higher than this caused excessive breakdown of the dicumene molecule and subsequent C deposition. Using a flow rate of one liter/min through the dicumene chromium, plating rates of 0.5 g/hr were realized.

Lower decomposition temperatures could be used if hydrogen iodide gas was added to the fluidized bed. This was done by bleeding HI at the rate of 0.05 liter/min into the gas stream as shown in figure 2. The HI then mixed with the dicumene chromium vapor in the fluidized bed and catalyzed the decomposition reaction. Temperatures as low as  $285^\circ\text{C}$  could be used for chromium deposition when using HI. The resulting coatings, however, contained objectionably high iodine contents. The iodine was present as chromium iodide,  $\text{CrI}_3$ . The iodine content varied between 5 and 15 wt percent depending on slight changes in the HI flow rate.

Typical chromium coatings produced without using the HI catalyst contained approximately 3 wt percent C. Using both hydrogen gas and HI catalyst with a HI-to-dicumene chromium ratio of 1:4, the carbon content was reduced to 0.3 wt percent (5).

Electron micrographs of agglomerated chromium coated MgO particles produced by this process without HI are shown in figures 4 and 5. The distribution of the dispersoid particles is good. The chromium platings consisted almost entirely of amorphous material as determined by x-ray examination. When the coated particles were heated to  $800^\circ\text{C}$ , the amorphous deposit crystallized and was analyzed to be  $\text{Cr}_{23}\text{C}_6$  by x-ray diffraction. No metallic chromium lines were observed.

## Molybdenum Deposition

The molybdenum was deposited by passing hydrogen and argon in a 1:1 ratio through a pot of molten molybdenum pentachloride,  $\text{MoCl}_5$ , heated to  $170^\circ\text{C}$ . The vapor and carrier gases then passed through feeder lines at  $200^\circ\text{C}$  and were decomposed in the fluidized bed at  $575^\circ\text{C}$ . By using a flow rate of 0.8 liter/hr through the  $\text{MoCl}_5$ , plating rates of 0.2 g/hr were realized.

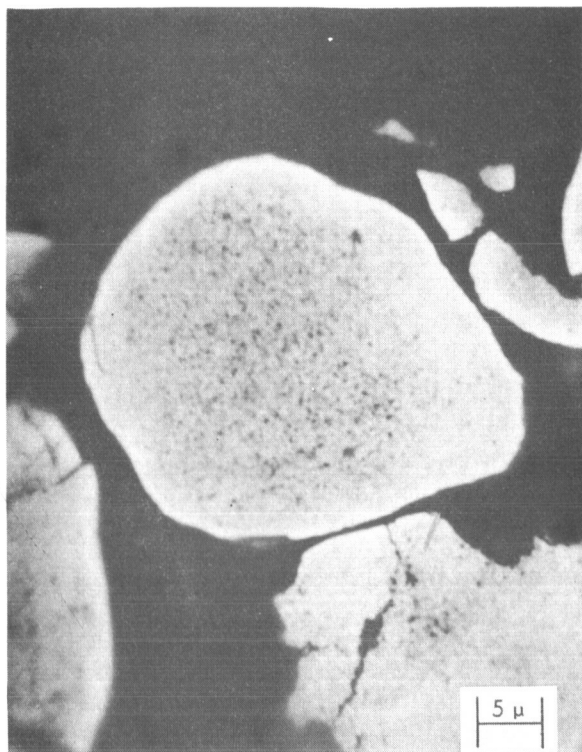


Figure 4. Electron Micrograph of Cr-32% MgO Composite Particle



Figure 5. Electron Micrograph of Cr-32% MgO Particle

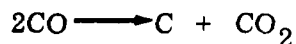
## Multiple-Element Deposition

The individual elements had to be vapor deposited in the fluidized bed in the order of decreasing precursor compound decomposition temperatures (Mo, Cr, Ni). A temperature 50° C above the deposition temperature of Cr (325° C) and Ni (190° C) caused agglomeration of the individual coated particles precluding uniform coating and dispersoid distribution.

## RESULTS AND DISCUSSION OF RESULTS

### Nickel Deposition

Three runs were made to determine the parameters for nickel deposition. Parameters and results are shown in table 1. The nickel carbonyl generator was maintained at 125° to 165° C. For optimum formation of nickel carbonyl the generator was operated at 125° C. A fluidized bed temperature between 220° and 185° C was used. The use of decomposition temperatures above 210° C caused excessive carbon deposition owing to the decomposition of the carbon monoxide



Decomposition temperatures below 190° C resulted in very low nickel deposition rates owing to apparent incomplete nickel carbonyl decomposition. Under the best conditions (generator 125° C; flow rate 1 liter/min each of carbonyl plus CO, and of argon; bed at 195° - 200° C), plating rates of 0.5 g/hr were realized.

Figure 6 is an electron micrograph of the material produced during run 3. The distribution of the oxide dispersoid particles in the nickel matrix appears to be good.

### Molybdenum Deposition

Eight experimental runs were conducted in which molybdenum was plated on zirconia particles. Two runs were stopped after the molybdenum deposition. The other six runs were plated also with chromium or/and nickel. Agglomeration of the fine  $\text{ZrO}_2$  particles was observed to occur at the  $\text{MoCl}_5$  decomposition temperature of 575° C. Figure 7 is a photomicrograph of the material produced by plating molybdenum on  $\text{ZrO}_2$  for 28 hrs. The powder obtained from the collapsed fluidized bed was pressed in a 3/8-inch-diameter die at 100,000 psi and vacuum sintered at 1500° C for 1 hour prior to metallographic preparation. Chemical analysis showed the sample contained 8.9 percent Mo. The molybdenum and the zirconia are segregated. The metallic particles in this structure contained no zirconia particles



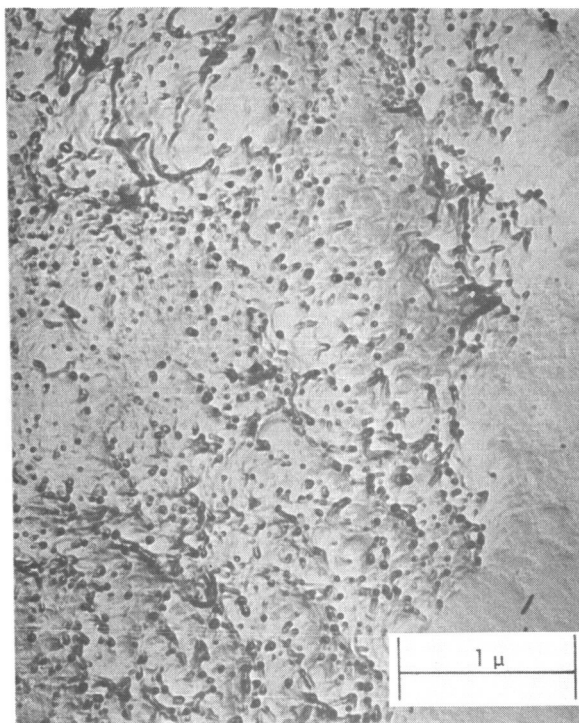


Figure 6. Electron Micrograph of Sintered Ni - ZrO<sub>2</sub> Particles Produced by Vapor Plating in Run No. 3, Table 1

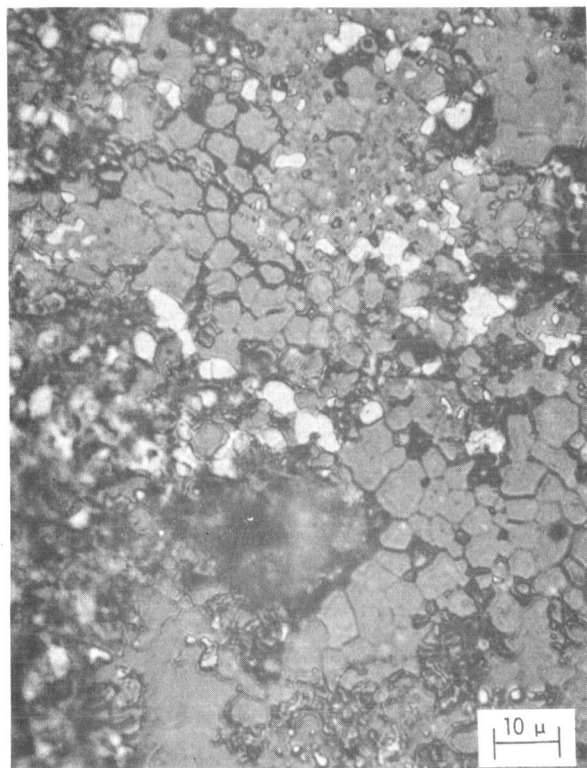


Figure 7. 8.9 Percent Molybdenum-ZrO<sub>2</sub> After Pressing and Sintering at 1500° C for 1 Hour

TABLE I  
RESULTS OF VAPOR PLATING NICKEL ON SUBMICRON  
OXIDE PARTICLES

Run Number	1	2	3
Dispersoid	$\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$	$\text{ZrO}_2$
Particle size ( $\mu$ )	0.05	0.05	0.10
Wt. dispersoid (g)	7.76	7.65	5.60
$\text{Ni}(\text{CO})_4$ generator temperature ( $^{\circ}\text{C}$ )	55	60	125
Fluidized bed temperature ( $^{\circ}\text{C}$ )	220	200	200
Plating Time (hr.)	5.5	5.0	18
Recovered Wt. (g)	9.01	10.12	9.00
Chemical Analysis			
% Ni	20.9		30.1
% C			0.34
% S	0.010		0.026

when examined by electron microscopy. The zirconia particles apparently agglomerated in the fluidized bed before plating took place. Molybdenum deposition then occurred in the presence of the larger agglomerated particles. During the vacuum sintering at 1500° C, the molybdenum subsequently agglomerated into regions as shown in figure 7.

### Multiple-Element Depositions

Five runs were made in which first molybdenum and then one other element (Cr or Ni) were deposited. These dual-element runs were all terminated before completion because of plugging of the reactor or excessive blowover. Plugging was caused by agglomeration of the particles and resultant fluidized-bed collapse and/or condensation of the precursor compound in the lines near the base of the fluidized bed or in the "T" section. The latter was usually caused by a lowering of the temperature in these parts. Temperature control of the system was difficult owing to the inherent instability of the process and the slow response of the system heaters.

One successful attempt was made to produce a triple-element alloy entirely by vapor plating. The elements were deposited in the order of molybdenum, chromium, and nickel. Using the optimum plating conditions previously described, plating times of 6, 16, and 16 hours were used respectively for Mo, Cr, and Ni. The resultant alloy contained 11.0 percent Mo, 35.9 percent Cr, 12.0 percent Ni, 5.2 percent C, and 35.9 percent ZrO<sub>2</sub> by difference. While it was possible to deposit nickel on zirconia as described in section 4.1 with only a 0.34 percent carbon content, this run produced a higher carbon content. This high carbon content made metallographic examination of the product difficult. The oxide particles clumped as the one shown in figure 8 in material taken from a later run.

Because the high carbon content was apparently related to the depositing of nickel, an attempt was made to add the nickel directly in the form of fine powder. Sheritt Gordon NF-1M, 0.6- to 1.4-micron-diameter nickel powder was added in one run after the molybdenum and chromium had been vapor plated onto the zirconia particles. The nickel was added slowly to the top of the fluidized bed. Mixing was accomplished by the motion of the bed and was continued for 2 hours. The fluidized bed temperature was then raised to 750° C for 2 hours to permit sintering. Figure 8 shows a light micrograph of the product. The oxide phase had segregated into large particles 10 to 20 microns in diameter. This run contained 85.0 percent Ni, 3.4 percent Cr, 4.6 percent Mo, 0.2 percent C, 0.003 percent S and 6.7 percent ZrO<sub>2</sub> by difference.

In another run, the nickel powder was added after plating the molybdenum; the chromium was then plated on the mixture of molybdenum-coated ZrO<sub>2</sub> and nickel powder. Metallographic examination showed that the oxide particles were segregated as in the previous run. This run contained 85.0 percent Ni, 3.4 percent Cr, 4.6 percent Mo, 0.21 percent C, and 6.8 percent ZrO<sub>2</sub> by difference.



Figure 8. Large Clump of Zirconia in Ni-Cr-Mo Alloy

## CONCLUSIONS AND RECOMMENDATIONS

Vapor plating fine oxide particles in a fluidized bed has produced nickel or chromium with apparently good oxide particle distribution. Samples so produced contain a high volume fraction of dispersoid ( $> 40$  percent), but are characterized by high carbon content.

Fine zirconia particles (0.1-micron diameter) at the temperature necessary for molybdenum pentachloride reduction,  $575^{\circ}\text{C}$ , agglomerated and sintered into 25- to 50-micron-diameter particles. Unless molybdenum can be deposited at a lower temperature, it seems impossible to produce dispersion-strengthened molybdenum or any alloy containing molybdenum with a uniform dispersoid distribution by the fluidized-bed technique. While these experiments were conducted on zirconia particles, it is reasonable to suspect that other oxides will similarly agglomerate during molybdenum vapor deposition.

A possible solution to the molybdenum deposition problem would be the use of molybdenum carbonyl,  $\text{Mo}(\text{CO})_6$  which could be decomposed as low as  $400^{\circ}\text{C}$ . But this process has an inherently high resultant carbon content.

The fluidized-bed process is capable of producing alloys of good dispersoid particle size and distribution provided that the precursor compounds can be applied at relatively low temperatures. Other methods of plating the dispersoid with metals, such as evaporation, sublimation and exploding wires of the elements, might be used inside the fluidized bed. These methods require no external heating of the fluidized-bed particles, thus helping to prevent agglomeration and sintering. Another advantage of using elemental sources would be the elimination of impurities that are picked up from the decomposing precursor compounds.

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